

## PATENT ABSTRACTS OF JAPAN

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## 54) METHOD FOR MANUFACTURING SUBSTRATE FOR MAGNETIC RECORDING MEDIUM

## 57)Abstract:

PROBLEM TO BE SOLVED: To provide a method of manufacturing a substrate for a magnetic recording medium having a NiP electroless plating layer with high adhesion to a glass substrate.

SOLUTION: After a glass substrate surface is lapped by using a rubbing liquid composition containing silicon carbide particles or alumina particles a maximum particle size of which is 130  $\mu\text{m}$  or smaller, a particle size is 110 nm or smaller at a 3% cumulative height, and a 0.5  $\mu\text{m}$  or larger at a 94% cumulative height, it is subjected to chemical etching. And then, it is subjected to NiP electroless plating.

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Claim(s)]

Claim 1] The manufacture method of the substrate for magnetic-recording media which a maximum droplet size is 10 micrometers or less, and is characterized by performing chemical etching processing and subsequently performing electroless deposition processing on it after the particle size in 3% of accumulation height performs lap processing on a glass-substrate front face using the polish liquid constituent with which the particle size is 110 micrometers or less and 94% of accumulation height contains a silicon-carbide particle or an alumina particle 0.5 micrometers or more.

Claim 2] The manufacture method of the substrate for magnetic-recording media according to claim 1 that a glass substrate is characterized by the bird clapper from SiO<sub>2</sub>-Li<sub>2</sub>O system glass ceramics.

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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

[001]

The technical field to which invention belongs] this invention relates to the manufacture method of the glass substrate for magnetic-recording media. Furthermore, it is involved in the manufacture method of a glass substrate and the substrate for magnetic-recording media which has a NiP electroless deposition layer with high adhesion. Specifically, it is related with the manufacture method of the substrate in high recording density magnetic-recording media, such as a film magnetic-recording disk of the cover half used by the information industry etc.

[002]

[Description of the Prior Art] In recent years, many hard disk equipments are used as external storage of information processors, such as a computer. After forming a NiP electroless deposition layer in the front face of the nonmagnetic substrate which consists of an aluminium alloy generally and performing necessary data smoothing, texture ring processing, etc., the magnetic disk carried in this hard disk equipment forms a non-magnetic metal ground layer, a magnetic layer, a protective layer, a lubricating layer, etc. one by one on it, and is produced.

[003] In the magnetic disk unit, although the head for record reproduction is moving by the fixed flying height in the magnetic-recording medium top, this flying height is very small with the rapid increase in the field recording density of magnetic-recording medium in recent years. Moreover, in order for the miniaturization of a magnetic disk unit and lightweight-ization to also progress quickly and to correspond to these, it is required to make still smaller granularity of the front face of a magnetic-recording medium, and medium surface roughness is already small to about several angstroms by Ra. Furthermore, since the shock resistance required of a magnetic disk since it corresponds to portability type hard disk equipment is also becoming 400G-800G, and a high value, in the substrate which consists of the conventional aluminium alloy to shock resistance, correspondence is difficult. Then, the glass substrate which can obtain very small surface roughness and is excellent also in the mechanical strength instead of the aluminium alloy substrate from standpoints, such as shock resistance and surface smooth nature, is beginning to be used.

[004] In many cases, in the aluminium alloy substrate which gave NiP electroless deposition, the concentric circle-texture ring is given to the substrate circumferencial direction by polish on the front face. If good, this mainly uses the friction property between the head for record reproduction, and a magnetic-recording medium, and aims at securing endurance. Moreover, in recent years, to replace with the texture ring by polish and to form a salient only in a SS zone with the texture ring by the laser beam, i.e., a laser beam, in connection with the flying height of the head at the time of a magnetic-disk-unit operation being remarkably small, is tried. (JP,8-129749,A etc.)

[005] However, since the salient configuration controllability is bad, it is very difficult unlike the aluminium alloy substrate which gave NiP electroless deposition, to irradiate a direct laser beam and to form a salient in a glass plate. Then, in order to apply laser texture technology to a glass substrate, it is necessary to form a NiP electroless deposition layer on a substrate beforehand.

[006] Forming a NiP electroless deposition film on a glass substrate is proposed by JP,61-54018,A. However, it is technically difficult to form a NiP layer in a glass substrate with sufficient adhesion by the electroless deposition method. Then, in order to improve the adhesion of a glass substrate and a NiP electroless deposition film, the method of split-face-izing mechanically or chemically the glass-substrate front face used for plating and the method of performing pretreatment of electroless deposition are proposed. for example, -- as the mechanical split-face-ized method -- aluminum 2O3 etc. -- the method which 100A or more of surface roughness grinds by Ra in center line average coarseness by the grinding stone using the abrasive material is learned, and as the chemical \*\*\*\*-ized method, after carrying out alkaline degreasing, the method of \*\*\*\*\*ing by the hydrofluoric acid etc. is learned Moreover, the method continuously carried out sensitization and activated with the solution of a palladium chloride with the solution of a stannous chloride as pretreatment of electroless deposition is proposed. (JP,7-272263,A etc.)

007]

problem(s) to be Solved by the Invention] However, by these methods, the NiP layer which has sufficient adhesion and sufficient smooth nature to obtain a good magnetic disk was not able to be formed by the electroless deposition method on the glass substrate. this invention is made in view of an above-mentioned point, and the purpose is excellent the adhesion of a glass substrate and a NiP electroless deposition layer, and it has high shock resistance and surface smooth nature, and is in moreover offering the manufacture method of the substrate for magnetic-recording media that a low surfacing height of a head is obtained by being stabilized.

008]

Means for Solving the Problem] As a result of inquiring wholeheartedly in view of the above-mentioned actual condition, by performing wrapping processing which used the specific polish abrasive grain for the glass substrate, this invention persons find out that the outstanding NiP layer which satisfies many above-mentioned requirements is formed on a substrate, and reach this invention. That is, after the summary of this invention performs lap processing on glass-substrate front face using the polish liquid constituent containing a silicon-carbide particle or an alumina particle with the maximum droplet size of 130 micrometers or less, a particle size [ in 3% of accumulation height / of 10 micrometers or less ], and a particle size [ in 94% of accumulation height ] of 0.5 micrometers or more, it performs chemical etching processing and consists in the manufacture method of the substrate for magnetic-recording media characterized by subsequently performing NiP electroless deposition processing.

009] Hereafter, this invention is explained in detail. After carrying out lap processing of the front face of a glass substrate, a detailed crevice is made to form in a substrate front face by carrying out chemical etching, in order that the substrate for magnetic-recording media of this invention may raise the adhesion of a glass substrate and a NiP electroless deposition layer. As a glass substrate, although especially the quality of the material is not limited, glass ceramics are desirable and the glass ceramics of a SiO<sub>2</sub>-Li<sub>2</sub>O system are used further suitably. Without spoiling surface smooth nature to some extent, since the amorphous field on the front face of a substrate can be alternatively changed if glass ceramics are used, since this can form a detailed crevice appropriately, it is suitable. When the degree of crystallization is small, a detailed depression is not formed of uniform etching, but adhesion tends to get worse by it.

010] The polish liquid constituent used for lap processing of this invention contains a silicon-carbide particle or an alumina particle as an abrasive grain, and particle size [ in / 110 micrometers or less and 94% of accumulation height / the particle size in 130 micrometers or less and 3% of accumulation height ] is required for the maximum droplet size of this abrasive grain, when that it is 0.5 micrometers or more raises the adhesion of a glass substrate and a NiP deposit. Each of maximum droplet sizes of these abrasive grains, 3% of accumulation height, and particle size in 94% JIS by the electric resistance examining method. R It asks by measuring by 6111. Moreover, it is desirable to use the liquid containing a surfactant as a polish liquid constituent of this invention.

011] Lap processing performed using the polish liquid constituent of this invention can be performed by the conventional method. As a typical method, the method of grinding a glass-substrate front face etc. is mentioned, for example, supplying the polish liquid of this invention to an abrasive cloth or polish putt, after performing thickness adjustment of glass by the grinding processing by the bonded abrasive. Although the glass substrate used does not need to be carried out even if the mirror finish of the front face is beforehand carried out by polish processing, in industrial production, the method of not performing polish processing in the meaning which reduces a process is suitable for it.

012] As for the glass substrate obtained by lap processing by this invention, it is desirable that center line surface roughness is 0.01-1.0 micrometers, and further 0.01-0.5 micrometers is suitable for it. This configuration contributes to formation of the detailed crevice formed by the chemical etching processing performed at a next process greatly. The glass substrate used for this invention has desirable glass ceramics, and it is suitable to use SiO<sub>2</sub>-Li<sub>2</sub>O system glass ceramics especially.

013] Subsequently chemical etching processing is performed to the glass substrate which performed lap processing. As an etching reagent used for chemical etching, fluorine acid system etching reagents, such as fluorine acid, a potassium fluoride, an ammonium fluoride, and an acid ammonium fluoride (NH<sub>4</sub>F-HF), are desirable. This will depend glass ceramics on the portion which \*\*\*\*\*s comparatively alternatively and the amorphous (amorphous-izing) portion of glass ceramics is crystallizing tending [ comparatively ] to remain, if etching processing is performed by the fluorine acid system etching reagent. It is thought that this portion that remained becomes the detailed crevice which brings about the anchor effect which was excellent when NiP electroless deposition was performed by this.

014] The size of this detailed crevice can be controlled by choosing suitably the concentration of an etching reagent, processing temperature, the processing time, etc. According to this invention, the glass substrate which has the detailed crevice whose width of face the length of a crevice is 4-20 micrometers, and is 1-5 micrometers on a front face can be obtained by lap processing and chemical etching processing. When the length and width of face of a crevice exceed the range of under the above-mentioned range, or this range, adhesion with a NiP electroless deposition layer is not obtained

ough. Here, the length of a crevice indicates a part for the longest bay in a direction perpendicular to the length to be depth of face for a part for a longest crevice bay. Moreover, when the length of each crevice differs from width of face, the 20 or more averages be the length of a crevice, and width of face.

015] Moreover, it is suitable for the aspect ratio of a crevice that it is 0.3-0.7. Here, an aspect ratio shows the maximum width of a hole, and the ratio (maximum width / the maximum length) of the maximum length. NiP seldom comes to enter at the time of plating as an aspect ratio is less than 0.3. Moreover, an anchor effect becomes weak and is not desirable if 0.7 is exceeded. Furthermore, when it observes by 600 times of SEM, as for the rate of area of a crevice, it is desirable that it is 0.5 - 50% to a substrate front face. The adhesion of plating will tend to become weak if the rate of area exceeds less than 0.5% and 50%.

016] The crevice on these front faces of a substrate is observed and measured as a black portion with the secondary electron image of a scanning electron microscope (SEM). That is, since a shadow is hit when it observes by the secondary electron image of SEM, a crevice is observed black. Specifically, using SEM 600 times the scale factor of 1000, the length and width of face of a crevice lean a substrate front face 40 degrees to the detector of a secondary electron line, observe it, and are measured.

017] The substrate for magnetic-recording media obtained by this invention is manufactured through a susceptibility-ized process, an activation process, and a NiP electroless deposition process one by one by the well-known method. And before susceptibility processing, degreasing processing is usually prepared. Moreover, it is good to establish a rinsing process between each process, to use ion exchange water or ultrapure water suitably as a wash water, and to check in a wash bath suitably.

018] A degreasing process is a process which washes the front face of a glass substrate, for example, the method of using ultrapure water, an alkali cleaner, an acid cleaning agent, a surfactant, etc. is mentioned. A susceptibility-ized process and an activation process are processes which give catalytic activity required in order to make a glass substrate start NiP electroless deposition. That is, a glass front face needs to form the catalyst nucleus of noble metals, such as Cu, Pt, Pd, and Ag, on the surface of glass, in order to start electroless deposition, since there is no catalytic activity.

019] Each above-mentioned process is carried out as follows by the well-known method. The divalent metal ion which consists of Sn, Ti, Pd, Hg, etc. is made to adsorb first in a susceptibility-ized process. Usually, it is used suitably, and in ordinary temperature, the tin chloride solution of about 0.05 g/l is immersed about 2 minutes into tin chloride solution, and rinses. Next, a catalyst nucleus is made to form in the included activation solution containing the noble metals which serve as the aforementioned catalyst nucleus as an activation process on the surface of a glass substrate / reduction operation of the divalent metal ion which was immersed and adsorbed the above-mentioned glass substrate. Usually, it is used suitably, and in ordinary temperature, the palladium-chloride solution of about 0.05 g/l is immersed about 2 minutes into palladium-chloride solution, and rinses. A susceptibility-ized process and an activation process are good also as the same process by using the mixed-water solution of tin chloride and a palladium chloride.

020] NiP electroless deposition of the glass substrate processed at the activation process is carried out by the well-known method. Usually, a commercial NiP electroless deposition bath is used and predetermined-time processing of the glass substrate is carried out in a plating bath. Although NiP electroless deposition layer thickness is chosen arbitrarily, for a good magnetic-recording medium, the range of 1-10 micrometers is good.

021] According to this invention persons' knowledge, in order to raise the adhesion of a glass substrate and a NiP layer, it is required to heighten a physical anchor effect and it is effective. That is, the length which was formed in the substrate front face by chemical etching processing according to this invention is considered that 1-5 micrometers [ 3-20 micrometers and width of face ], Sn enters [ an aspect ratio ] by reception-ized processing, and Pd enters [ a reduction operation by activation further in the detailed crevice of 0.3-0.7. Therefore, since a NiP film is formed into this detailed hole in case a NiP film is formed by NiP plating processing, a physical anchor effect is heightened and it is thought that adhesion with a glass substrate and a NiP deposit is strengthened by this. If needed, polish processing can be performed on the glass substrate which gave NiP electroless deposition can perform suitably texture processing of the texture ring by the laser beam, a machine texture ring, etc.

022] Subsequently, a magnetic-recording layer is formed according to a conventional method. Usually, it forms so that the laminating of each class may be carried out to the order of Cr ground layer, a magnetic layer, a protective layer, and a lubricating layer. Although the thickness of Cr ground layer is set as a magnetic-recording medium according to desired magnetic properties, it is usually 100-1000Å. Cr ground layer is usually pure -- although formed by Cr, you may make other elements to a 20 atom % grade contain in total Although there is usually one Cr ground layer, if it is a request, also let it be the multilayer which consists of two or more layers.

023] A magnetic layer is usually formed by Co system alloy, for example, CoNiCr, CoCr, CoCrTa, CoCrPt, CoCrPtTa, CoCrPtB, CoNiPt, CoNiCrBTa, CoSm, etc. The thickness of a magnetic layer is usually 100-500Å. You may be a multilayer even if the number of magnetic layers is also one. A protective layer is usually formed by metallic

des, such as carbon materials, such as amorphous carbon and hydrogenation carbon, and a silica, a zirconia, and 30-200 Å of the thickness is usually 30-200 Å preferably. You may be a multilayer even if the number of protective layers is also one.

[24] A lubricating layer is formed by applying a fluorine system fluid lubrication agent etc. to a protective layer. In addition, although a protective layer and a lubricating layer are not indispensable as a magnetic-recording medium, it is very desirable [ a lubricating layer ] to prepare both this layer, considering the endurance of a magnetic-recording medium, a friction property with the head for record reproduction, etc. Formation of a ground layer, a magnetic layer, and a protective layer can be performed by conventional methods, such as a DC-sputtering method, a RF-sputtering method, and a vacuum deposition method. According to this invention, by performing NiP electroless deposition to the above glass substrates, it has the adhesion of sufficient strength which does not cause exfoliation with a glass substrate and a NiP electroless deposition film etc., and it becomes possible to obtain the magnetic-recording medium excellent shock resistance.

[25] [example] Hereafter, although an example explains this invention still in detail, this invention is not limited to the following examples, unless the summary is exceeded. In addition, in each example, measurement and evaluation were performed on condition that the following.

[26] (1) Using the scanning electron microscope (SEM) 600 times the surface type-like scale factor of a glass substrate of this, it leaned 40 degrees, observed to the detector of a secondary electron line, and asked for the rate of area of the crevice to the length and the width of face, aspect ratio, and substrate front face of a crevice on the front face of a substrate.

(2) Adhesion JISK5400 of a glass substrate and a NiP electroless deposition layer The cross cut adhesion test of 8.15 estimated adhesion. It is shown that the evaluation mark 10 have good adhesion.

(3) With the surface roughness meter (KEERUE ten call company make P-12) which has the sensing pin whose nose of diameter is  $\phi 0.2$  micrometer, surface roughness center line surface roughness (Ra) was performed by 240 micrometers of measurement length, and calculated and evaluated the average.

[27] Glass ceramics of the  $\text{SiO}_2$ - $\text{Li}_2\text{O}$  system of example 1 marketing. After using it and performing grinding (lapping) processing by the bonded abrasive, it wrapped by grain-size partition #1000 (less than [ maximum-droplet-size 27 micrometer ], particle size of 23 micrometers or less of 3% of accumulation height, particle size of 5.0 micrometers or more of 94% of accumulation height) of the artificial abrasives F0 (a compound artificial emery, 3.90 or more specific gravity, more than aluminum  $2\text{O}_3$  : 45 % of the weight, less than [  $\text{TiO}_2$  : 2.0 % of the weight ], less than  $\text{ZrSiO}_4$  : 49 % of the weight ]) made from FUJIMIINKOPORE. The center line average surface roughness (Ra) of the obtained substrate was 0.3 micrometers.

[28] Next, it rinsed after the washing processing for 10 minutes at 50 degrees C of bath temperature by the alkaline cleaning agent for glass ( PK-LCG22 by , Inc. Parker), and rinsed by immersing for 2 minutes and the above-mentioned glass ceramics at a room temperature, and subsequently to the inside of acid ammonium-fluoride (  $\text{NH}_4\text{F}$  by Sinto chemistry incorporated company 4 F-HF JIS number K8817) 50 g/l, performing etching processing.

[29] The length of the detailed crevice on the obtained front face of a glass substrate was 4.9 micrometers, width of crevice was 2.9 micrometers, and 0.59 and the rate of area of the aspect ratio of a crevice were 13.4%. Next, it is the glass substrate which has a detailed crevice  $\text{SnCl}_2$  of commercial 0.05 g/l It rinsed by having been immersed in solution for 10 minutes at the room temperature, and susceptibility-ized processing was performed. Then,  $\text{PdCl}_2$  of commercial 0.05 g/l It rinsed by having been immersed in solution for 2 minutes at the room temperature, and activation was performed. Subsequently, the NiP layer of 15 micrometers of thickness was formed by NiP electroless deposition. Furthermore, lapping processing of 1 hour was performed at 150 degrees C. Thus, as a result of evaluating the adhesion of the obtained NiP layer and a glass substrate, evaluation mark are 10 and having good adhesion was checked.

[30] Abrasive material FO#1000 of example 2 example 1. instead of using it -- FO (the compound artificial emery made from FUJIMIINKOPORE --) 3.90 or more specific gravity, aluminum  $2\text{O}_3$  : 45 % of the weight or more, less than [  $\text{TiO}_2$  : 2.0 % of the weight ],  $\text{ZrSiO}_4$  : Except having wrapped by grain-size partition #2000 not more than 49 % of the weight (15 micrometers or less of maximum droplet sizes, particle size of 14 micrometers or less of 3% of accumulation height, particle size of 2.0 micrometers or more of 94% of accumulation height), it is the same method as in example 1, and the NiP electroless deposition layer was formed in the glass substrate. The centerline surface roughness after lap processing, the configuration of the detailed crevice after etching processing, and the adhesion of a glass substrate and a NiP electroless deposition layer are shown in Table 1. All are 10 and the evaluation mark of adhesion have good adhesion for them.

[31] Instead of using abrasive material FO#1000 of example 3 example 1, except having wrapped by GC#3000 (the product made from FUJIMIINKOPORE, 96 % of the weight or more of  $\text{SiC}$ (s), 13 micrometers or less of maximum

droplet sizes, particle size of 11 micrometers or less of 3% of accumulation height, particle size of 2.0 micrometers or more of 94% of accumulation height), it is the same method as an example 1, and the NiP electroless deposition layer was formed in the glass substrate. A result is shown in Table 1.

032] Instead of using abrasive material FO#1000 of example of comparison 1 example 1, except having wrapped with cerium-oxide polish liquid (speed FARM company make, 10 micrometers or less of maximum droplet sizes, particle size of 0.24 micrometers or less of 3% of accumulation height, particle size of 3.0 micrometers or more of 94% of accumulation height), it is the same method as an example 1, and the NiP electroless deposition layer was formed in the glass substrate. A result is shown in Table 1. The evaluation mark of adhesion are 5 and were not able to obtain sufficient adhesion.

033]

Table 1]

	Center line	Concave Section	Adhesion	Surface roughness	Length	Width of face	Aspect
Ratio	Rate of area	Evaluation mark (micrometer)	(micrometer)	(micrometer)	(%)		
example 1.	0.3	4.9	2.9	0.59	13.4	10	Example 2 0.05 3.4 2.1 0.62 2.7 10 Example 3 0.09
1 1.5 0.48 1.3 10 Example 1 of comparison	0.0005	3.4	3.3	0.97	0.3	5	-----.

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